[Second Edition.]

PATENT SPECIFICATION

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PROVISIONAL SPECIFICATION

Improvements in the Production of Artificial Filaments, Films, Foils and like Materials.

I, HENRY DREYFUS, a British subject, of Celanese House, 22 & 23, Hanover Square, London, W.1, do hereby declare the nature of this invention to be as follows:—

This invention relates to the production of artificial materials and particularly to the spinning or extrusion of filaments, films, foils and like materials from molten organic filament-forming compositions.

In the spinning or extrusion of molten organic filament-forming compositions, difficulties can arise from the fact that the melting point of the composition is only slightly below the temperature at which the composition decomposes and from the fact that the composition, whether solid or molten, is a poor conductor of heat. The first of these facts limits the time during which it is permissible for the composition to be exposed to the temperature necessary to melt it while the second makes it difficult to melt the composition in the limited time available and to avoid locally heating the composition to a temperature above that at which the composition decomposes. Furthermore, the compositions when melted are apt to be very viscous unless heated to such a temperature that they are liable to decompose, and difficulty can be experienced while initiating the spinning operation in priming or filling with the molten composition the pump or like means employed for extrusion.

It has now been found that in the spinning of molten organic filament-forming compositions, the melting of the compositions can be conveniently and efficiently effected by immersing the composition in a liquid heating medium which thus surrounds the solid composition so that heat is applied to the whole surface thereof and rapid and complete melting of the composition is achieved. If, moreover, the heating liquid is of lower specific gravity than the composition to be melted and extruded, this liquid can very conveniently serve for priming the pump or like extruding means, the composition to be extruded then entering the pump without difficulty as soon as it begins to be melted under the influence of the heating liquid.

In the spinning or otherwise shaping by extrusion of a molten organic filament-forming composition, therefore, the composition can be fed to the point from which it is extruded through a body of liquid that is of lower density than said composition and is maintained at a temperature sufficiently high to melt the composition, so that the composition reaches said point in a molten condition. The molten composition collects below the heating liquid and can readily be drawn off and extruded. Moreover the extrusion of the molten composition can readily be initiated by allowing the heating liquid to be extruded in the first instance, the extrusion of the filament-forming composition commencing when the composition becomes available in the molten state by melting of the solid composition in the heating liquid. The extrusion operation can thus be started without difficulty notwithstanding a high degree of viscosity in the molten composition.

The heating liquid employed should naturally be such as to have no deleterious effect, whether by chemical or physical action, on the composition to be melted or extruded. In particular, the liquid should be stable at the temperatures employed for melting the composition so that it does not decompose and give rise to products which might have an undesirable effect on the composition. As the liquid is of lower density than the composition to be extruded, it is generally convenient to employ an organic substance, which may be either liquid or solid at ordinary temperatures but is liquid at the temperature required for melting the material to be extruded. Thus, for example, where temperatures of the order of 200—300° C. are required and where the composition to be extruded has a specific gravity a little over unity, stearyl alcohol or other fatty alcohols containing at least 12 carbon atoms may be employed. Alternatively, hydrocarbons, particularly aliphatic hydrocarbons, and similar organic compounds of low gravity and containing a large number of carbon atoms, e.g. 30—40, may be used.

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Filament-forming composition that is still in a solid state may be restrained from reaching the point from which the composition is to be delivered for extrusion by means of a grid or sieve disposed a short distance above said point and adapted to retain unmelted pieces of the composition to keep them under the melting influence of the heating liquid for a longer period.

The heating liquid through which the composition is passed may be contained in a vessel in which provision may be made for an inert atmosphere to be maintained over the surface of the liquid, e.g. by supplying nitrogen under slight pressure to the space in the vessel above the liquid. In order to feed the solid composition through the liquid, a lock or chamber may be provided through which the composition may be dropped, piece by piece, to the surface of the liquid through which it passes by gravity, the lock or chamber serving not only to maintain the pressure above the liquid in the vessel, but also to prevent undue loss of heat by way of the opening through which the composition is passed into the vessel. On sinking through the liquid the composition, or such part of it as may still be solid, is retained by a sieve or grid below the surface of the liquid through which the composition does not pass until it is molten. On passing through the sieve or grid the molten composition forms a reservoir or pool at the bottom of the vessel from which material is drawn for the purpose of spinning or extrusion. Thus the molten composition may be extruded by means of a constant delivery pump whose inlet communicates directly with the bottom of the vessel. Means may be provided to maintain the quantity of molten composition available in the melting vessel by employing the level of the heating liquid as a control. Thus, a float may be provided on the surface of the heating liquid and the rise and fall of this float may make and break an electrical contact by means of which the pressure lock is operated and pieces of solid composition automatically fed through it. Alternatively, the making or breaking of an electrical contact in this manner may serve to give a visual indication of the amount of composition available, the further supply of solid composition being controlled manually in accordance with such visual indication.

The apparatus is preferably arranged so that the quantity of molten composition available for extrusion at any time is a minimum consistent with the maintenance of a constant supply. In this way, the period during which the composition is exposed to the temperature necessary to melt it is further reduced. For reasons of lightness and economy, it is also desirable that the body of heating liquid should be as small as possible having regard to its function as a reservoir of heat from which heat is drawn to melt the composition employed. If desired, the apparatus may be arranged so that two or more pumps may be fed from a single pool or reservoir of molten composition.

The heating of the heating liquid may be effected by any convenient means according to the temperature to be maintained and other relevant considerations. Thus, the vessel in which the composition is melted may be enclosed in a jacket containing a heating fluid or it may be surrounded with electrical windings. Or again, the liquid heating medium may be heated in a separate vessel and circulated through the vessel in which the composition is melted or through several such vessels. The whole apparatus is preferably externally lagged so as to prevent loss of heat and discomfort to the operatives. Whatever means are employed for the application of heat to the apparatus the heat should be supplied to every part of the apparatus containing the heating liquid or the molten composition. Thus, it is convenient for the vessel containing the heating liquid and the lock or chamber through which the solid composition is fed, and also the pump by means of which the molten composition is extruded, to be enclosed together as a single heat-insulated unit. The heat capacity of the unit may be augmented by including in its structure large bodies of metal. Thus, for example, the spinning or extruding pump may be surrounded by a heavy block of metal or by a mass of metal turnings or filings so as to provide a reservoir of heat to prevent undesirable fluctuations in temperature.

Control of the heat supplied to the apparatus can be effected by suitable temperature regulating means in accordance with the temperature existing within the apparatus. While, as mentioned above, it is desirable to supply heat to all parts of the apparatus containing liquid heating medium or molten composition, separate sources of heat under separate temperature controls may be employed for the different parts, e.g., one control for the melting vessel and its supply lock and another for the pump and the passage by which molten composition is fed to it. Again, the supply of heat to the melting vessel may be effected by circulation of heating liquid separately heated, and controlled by controlling the circulation, while the heating of the pump and the

control of that heating may be effected electrically.

The invention is applicable generally to the spinning or extrusion of organic filament-forming compositions that may be spun in the molten state and is particularly advantageous where, as is commonly the case with such compositions, the compositions are liable to decompose or be otherwise injuriously affected at temperatures not much exceeding those necessary to melt them. Examples of such materials are the synthetic linear superpolyamides made, e.g. by the condensation of diamines with dicarboxylic acids. Or again, the invention may be applied to compositions having a basis of cellulose derivative such as an organic ester or mixed ester of cellulose, e.g. cellulose acetate, cellulose propionate or cellulose butyrate cellulose acetate-butyrate cellulose acetate-propionate and cellulose acetate-stearate or cellulose ethers such as ethyl and benzyl cellulose.

Dated this 31st day of December, 1942.
STEPHENS & ALLEN,
Chartered Patent Agents,
Wykeham House, Gordon Avenue,
Stanmore, Middlesex.

COMPLETE SPECIFICATION

Improvements in the Production of Artificial Filaments, Films, Foils and like Materials.

I, Henry Dreyfus, a British subject, of Celanese House, 22 & 23, Hanover Square, London, W.1, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement :—

This invention relates to the production of artificial materials and particularly to the spinning or extrusion of filaments, films, foils and like materials from molten organic filament-forming compositions.

In the spinning or extrusion of molten organic filament-forming compositions, difficulties can arise from the fact that the melting point of the composition is only slightly below the temperature at which the composition decomposes and from the fact that the composition, whether solid or molten, is a poor conductor of heat. The first of these facts limits the time during which it is permissible for the composition to be exposed to the temperature necessary to melt it while the second makes it difficult to melt the composition in the limited time available and to avoid locally heating the composition to a temperature above that at which the composition decomposes. Furthermore, the compositions when melted are apt to be very viscous unless heated to such a temperature that they are liable to decompose, and difficulty can be experienced while initiating the spinning operation in priming or filling with the molten composition the pump or like means employed for extrusion.

It has now been found that in the spinning of molten organic filament-forming compositions, the melting of the compositions can be conveniently and efficiently effected by immersing the composition in a liquid heating medium which thus surrounds the solid composition so that heat is applied to the whole surface thereof and rapid and complete melting of the composition is achieved. If, moreover, the heating liquid is of lower specific gravity than the composition to be melted and extruded, this liquid can very conveniently serve for priming the pump or like extruding means, the composition to be extruded then entering the pump without difficulty.

According to the present invention therefore, in the spinning or otherwise shaping by extrusion of a molten organic filament-forming composition, the composition is fed to the point from which it is extruded through a body of liquid that is inert to the composition and is maintained at a temperature sufficiently high to melt the composition, so that the composition reaches said point in a molten condition. The liquid is preferably of lower density than the composition, so that the molten composition collects below the heating liquid and can readily be drawn off and extruded. Moreover the extrusion of the molten composition can readily be initiated by allowing the heating liquid to be extruded in the first instance, and then changing over to the extrusion of the filament-forming composition. The extrusion operation can thus be started without difficulty notwithstanding a high degree of viscosity in the molten composition.

The heating liquid employed should naturally be inert to the composition, i.e. such as to have no deleterious effect, whether by chemical or physical action, on the composition to be melted or extruded. In particular, the liquid should be stable at the temperatures employed for melting the composition so that it does not decompose and give rise to products

which might have an undesirable effect on the composition. As a liquid of lower density than the composition to be extruded, it is generally convenient to employ an organic substance, which may be either liquid or solid at ordinary temperatures but is liquid at the temperature required for melting the material to be extruded. Thus, for example, where temperatures of the order of 200—300° C. are required and where the composition to be extruded has a specific gravity a little over unity, stearyl alcohol or other fatty alcohols containing at least 12 carbon atoms may be employed. Alternatively, hydrocarbons, for example aliphatic hydrocarbons containing a large number of carbon atoms, e.g. 30—40, or mixtures thereof may be used. Thus paraffin wax can be employed.

The heating liquid through which the composition is passed may be contained in a vessel in which provision may be made for an inert atmosphere to be maintained over the surface of the liquid, e.g. by supplying nitrogen under slight pressure to the space in the vessel above the liquid. In order to feed the solid composition through the liquid, a lock or chamber may be provided through which the composition may be dropped, piece by piece, to the surface of the liquid through which it passes by gravity. On sinking through the liquid the composition, or such part of it as may still be solid, is retained by a sieve or grid below the surface of the liquid through which the composition does not pass until it is molten. On passing through the sieve or grid the molten composition forms a reservoir or pool at the bottom of the vessel from which material is drawn for the purpose of spinning or extrusion. Thus the molten composition may be extruded by means of a constant delivery pump whose inlet communicates directly with the bottom of the vessel. Means may be provided to maintain the quantity of molten composition available in the melting vessel by employing the level of the heating liquid as a control. Thus, a float may be provided on the surface of the heating liquid and the rise and fall of this float may make and break an electrical contact by means of which the pressure lock is operated and pieces of solid composition automatically fed through it. Alternatively, the making and breaking of an electrical contact in this manner may serve to give a visual indication of the amount of composition available, the further supply of solid composition being controlled manually in accordance with such visual indication.

The apparatus is preferably arranged so that the quantity of molten composition available for extrusion at any time is a minimum consistent with the maintenance of a constant supply. In this way, the period during which the composition is exposed to the temperature necessary to melt it is further reduced. For reasons of lightness and economy, it is also desirable that the body of heating liquid should be as small as possible having regard to its function as a reservoir of heat from which heat is drawn to melt the composition employed. If desired, the apparatus may be arranged so that two or more pumps may be fed from a single pool or reservoir of molten composition.

The heating of the heating liquid may be effected by any convenient means according to the temperature to be maintained and other relevant considerations. Thus, the vessel in which the composition is melted may be enclosed in a jacket containing a heating fluid or it may be surrounded with electrical windings. Or again, the liquid heating medium may be heated in a separate vessel and circulated through the vessel in which the composition is melted or through several such vessels. The whole apparatus is preferably externally lagged so as to prevent loss of heat and discomfort to the operatives.

Control of the heat supplied to the apparatus can be effected by suitable temperature regulating means in accordance with the temperature existing within the apparatus. While it is desirable to supply heat to all parts of the apparatus containing liquid heating medium or molten composition, separate sources of heat under separate temperature controls may be employed for the different parts, e.g. one control for the melting vessel and its supply lock and another for the pump and the passage by which molten composition is fed to it. Again, the supply of heat to the melting vessel may be effected by circulation of heating liquid separately heated, and controlled by controlling the circulation, while heating of the pump and the control of that heating may be effected electrically.

The invention is applicable generally to the spinning or extrusion of organic filament-forming compositions that may be spun in the molten state and is particularly advantageous where, as is commonly the case with such compositions, the compositions are liable to decompose or be otherwise injuriously affected at temperatures not much exceeding those necessary to melt them. Examples of such compositions are the substances known as the synthetic linear superpolyamides made, e.g. by the condensation of diamines with

dicarboxylic acids. Other compositions to which the invention may be applied are those consisting of or having a basis of a polyvinyl compound, e.g. polyvinylidene chloride or a copolymer of vinyl chloride with vinyl acetate. Or again, the invention may be applied to compositions consisting of or having a basis of cellulose derivative such as an organic ester or mixed ester of cellulose, e.g. cellulose acetate, cellulose propionate or cellulose butyrate, cellulose acetate-butyrate, cellulose acetate-propionate and cellulose acetate-stearate or cellulose ethers such as ethyl and benzyl cellulose.

By way of example, one form of apparatus in accordance with the present invention will now be described in greater detail with reference to the accompanying drawing, which is a sectional side elevation of the apparatus.

The apparatus comprises in general a melt chamber 1, a pump chamber 2 and a jet and filter assembly 3. The melt chamber and pump chamber are formed as cavities in a large cast iron vessel, 4 the pump chamber 2 being in the form of a plain cylindrical cavity in the bottom of the vessel and the melt chamber having a cylindrical upper portion and a conical lower portion and being disposed in the upper part of the vessel. The chamber 1 is closed by means of a heavy cover 5 held down by bolts 6 and sealed by means of a copper sealing ring.

Passing through a central pressure gland 8 in the cover 5 is a valve rod 9 having a screw portion 10 and a hand-wheel 11 by means of which it may be raised and lowered so that its lower end 12 may be raised from or engaged with the apex of the conical part of the chamber 1, which the end of the rod 9 is formed to fit. The apex of the conical part communicates by way of a passage 14 and connecting pipe 15 with the pump 16 in the pump chamber 2, and this communication may be opened and closed by raising or lowering the valve rod 9. The valve rod 9 is formed with an axial bore in which fits a further valve rod 17 passing into the valve rod 9 through a pressure gland 18 and having a screwed portion 19 and a hand-wheel 20, by means of which it may be raised and lowered. The lower end 21 of the valve rod 17 is pointed so as to fit on a seating 22 in the bore of the valve rod 9. Just above the seating 22 openings 23 in the wall of the valve rod 9 give access between the bore of the rod 9 and the melt chamber 1. A sieve or grid 24 extends across the chamber 1 near the bottom of the cylindrical portion thereof, being provided with a collar 25 which fits loosely round the valve rod 9 and with a flange 26 which fits the cylindrical portion of the chamber 1 and rests on the conical portion thereof.

A float 27 is provided in the chamber 1 from which a contact rod 28 extends upwardly through a guide plug 29, the bore of which is a loose fit round the rod 28. A second contact rod 30 passes through an insulating pressure gland 31 in the cover 5 so that as the float 27 rises and falls contact is made and broken between the rods 28, 30, thereby establishing or breaking an electrical connection between the wires 32, 33, connected respectively to the cover 5 and the contact rod 30.

During the operation of the device the melt chamber 1 contains a pool 35 of molten composition and a substantial body 36 of the liquid inert to the composition and of lower specific gravity. Pieces of solid composition 37 rest on the grid or sieve 24. The solid composition is introduced into the chamber 1 by means of a lock generally indicated at 38 comprising two plug valves 39, 40 in series, the plugs having discs 41, 42 respectively secured thereto whose edges have notches 43 each adapted to engage with the periphery of the other disc. By these means the valves 39 and 40 are interlocked so that neither can be opened unless the other is completely closed. The valves 39, 40 give access to an oblique passage 44 leading through the cover 5 to the chamber 1. In order to insert solid composition, the valve 39 is opened (the valve 40 being closed) and a piece of composition is dropped through the aperture 45 and is checked by the valve 40. The valve 39 is then closed and the valve 40 is opened, whereupon the piece of composition passes through the passage 44 into the chamber 1. The opening of the electrical connection between wires 32 and 33 gives an indication that fresh composition is required and the insertion of fresh composition raises the level of the liquid 36 in the chamber 1 so as to re-establish the contact until a further supply of fresh composition is required.

Heat is supplied to the melt chamber 1 by means of heating elements 47 supplied with current through leads 48, the heat so supplied maintaining the liquid 36 at such a temperature as to melt the solid composition 37 at the required rate. The solid composition sinks through the liquid 36 and rests on the grid or sieve 24 and, when molten, passes through the grid to join the pool of molten composition 35.

The spinning operation is started by melting an initial charge of composition, the valve 9 being closed by means of the hand-wheel 11 until a sufficient pool 35 of molten composition has accumulated.

While the pool 35 is accumulating, the valve 17 may be opened so that the liquid 36 may pass through the apertures 23 and through the bore of the valve rod 9 to the passage 14 and thence by way of the pipe 15 to the pump 16 by which it is forced through a filter 50 and jet 51. When a sufficient pool of molten composition 35 has accumulated, the valve 17 is closed (it is in this position that the apparatus is shown) and the valve 9 is opened so that the molten composition passes from the pool 35 through the pump 16 to the spinning jet 51, the pump and jet being already primed by means of the inert liquid 36 which has passed through them.

The action of the pump 16 is assisted by means of an atmosphere of nitrogen maintained in the chamber 1 above the surface of the liquid 36. This is supplied to the vessel by means of an oblique passage (not shown) similar in form to the passage 44 through which solid composition is supplied to the chamber. If desired the passage 14 may be connected directly to the filter and jet assembly 3, the pump 16 being omitted, and extrusion effected solely by the pressure of the atmosphere of nitrogen above the surface of the liquid 36.

The vessel 4 and the filter and jet assembly 3 are insulated against heat loss by means of a heavy layer 52 of a non-conducting material. The space in the pump chamber 2 surrounding the pump is filled with metal turnings 54 which augment the heat capacity of that part of the vessel 4 surrounding the pump chamber and help to maintain steady the temperature of the molten composition during its passage from the pool 35 to the spinning jet 51. In addition, heat may be supplied to the pump chamber 2, and also if desired to the filter and jet assembly 3 by means such as electrical heating elements 55 supplied with current through leads 56.

Having now particularly described and ascertained the nature of my said invention and in what manner the same is to be performed, I declare that what I claim is:—

1. Process for the extrusion of a fusible organic filament-forming composition, said process comprising feeding the composition to a point from which it is to be extruded through a body of heating liquid that is inert to said composition, maintaining said heating liquid at a temperature sufficiently high to melt the composition in its passage through said heating liquid, and extruding the molten composition.

2. Process for the extrusion of fusible organic filament-forming compositions comprising feeding the composition downwards to a point from which it is to be extruded through a body of heating liquid that is inert to and of lower density than said composition and is maintained at a temperature sufficiently high to melt the composition during its passage through the heating liquid, and extruding the molten composition.

3. Process according to Claim 2, comprising initiating the operation by melting an initial charge of composition, initiating the extrusion of said composition by first extruding heating liquid, and then changing over to extrusion of the molten composition.

4. Process according to any of the preceding claims comprising straining the composition while it is in the heating liquid so as to prevent the passage of solid material to the point of extrusion.

5. Process according to any of the preceding claims comprising maintaining over the surface of the heating liquid, an inert atmosphere under pressure.

6. Process according to any of the preceding claims comprising feeding fresh solid composition to the heating liquid in accordance with the level of the surface of said liquid so as to maintain said level substantially constant.

7. Process according to any of the preceding claims, wherein the heating liquid employed is stearyl alcohol or other fatty alcohol containing at least 12 carbon atoms.

8. Process according to any of the preceding claims 1—6, wherein the heating liquid is molten paraffin wax.

9. Process for the extrusion of a fusible organic filament-forming composition substantially as described.

10. Apparatus for the melting and extrusion of fusible organic filament-forming compositions, said apparatus comprising a melting vessel for containing a heating liquid inert to the composition, said vessel having an outlet for the withdrawal of melted composition, means for heating a liquid in said vessel, means for feeding the composition through said liquid to said outlet and means for withdrawing molten composition through said outlet and extruding said composition.

11. Apparatus according to Claim 10, comprising means operable from outside the vessel for closing the outlet of the vessel to the passage of molten composition during the melting of an initial charge of composition.

12. Apparatus according to Claim 11, comprising means for permitting the heating liquid to pass through the outlet of the vessel while preventing molten composition from doing so.

13. Apparatus according to any of the preceding Claims 10—12, comprising a grid or sieve extending across the vessel below the liquid level therein and in the path of the composition being fed to the point of extrusion.

14. Apparatus according to any of the preceding Claims 10—13, comprising means for supplying gas under pressure to the top of the vessel.

15. Apparatus according to any of the preceding Claims 10—14, comprising a spinning pump for the extrusion of the molten composition.

16. Apparatus according to any of the preceding Claims 10—15, comprising detector means adapted to give an indication outside the vessel of the level of heating liquid within the vessel.

17. Apparatus according to Claim 16, comprising a member within the vessel adapted to float on the surface of the heating liquid therein, an electric contact carried by said member, a stationary contact adapted to be engaged thereby, and an electric circuit adapted to be completed by the engagement of said contacts.

18. Apparatus according to Claim 16 or 17, comprising automatic means under the control of the detector means for supplying fresh solid composition to the vessel so as to maintain the level of the heating liquid within the vessel substantially constant.

19. Apparatus according to any of the preceding Claims 10—18, comprising separate heating means for the vessel and other parts of the apparatus containing liquid or solid composition, and separate means for controlling said heating means in accordance with the temperatures of the parts of the apparatus respectively heated thereby.

20. Apparatus for the extrusion of fusible organic filament-forming compositions substantially as shown in the accompanying drawings.

21. Apparatus for the extrusion of fusible organic filament-forming compositions substantially as described.

Dated this 22nd day of December, 1943.
STEPHENS & ALLEN,
Chartered Patent Agents,
Wykeham House, Gordon Avenue,
Stanmore, Middlesex.

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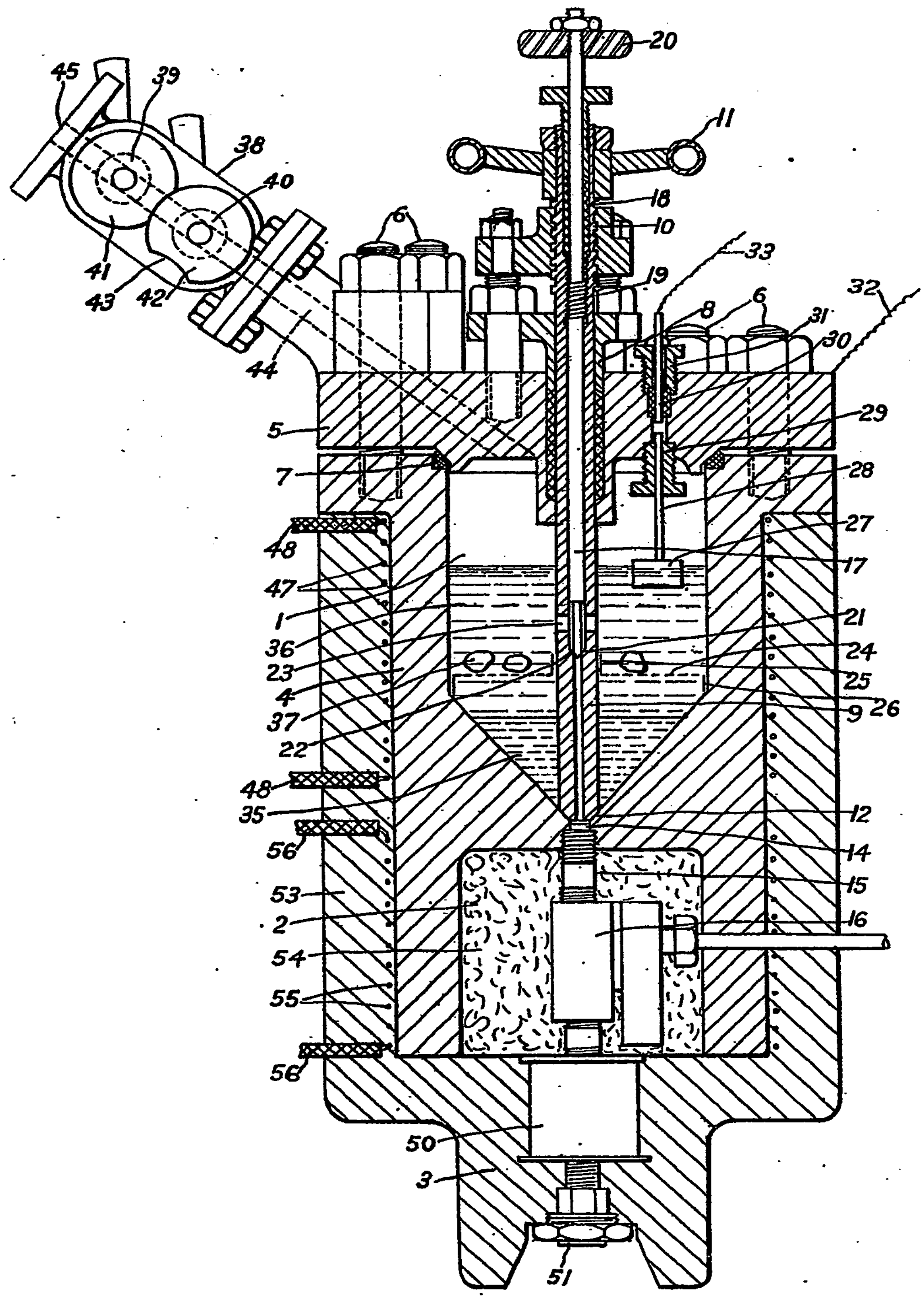
565,489 COMPLETE SPECIFICATION 1 SHEET

2nd Edition

[This Drawing is a reproduction of the Original on a reduced scale.]



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